

**ACTIVE CARBON AND METHOD FOR PRODUCING THE SAME**

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**Abstract of JP2001146410**

**PROBLEM TO BE SOLVED:** To provide a method for producing active carbon good in filling properties as an active carbon material and increasing the bulk density when converted into an electrode, to produce the active carbon and to provide the electrode for an electric double layer capacitor improved in capacity using the active carbon. **SOLUTION:** This active carbon is obtained by mixing a granular carbide in a spherical shape with a carbide having a smaller grain diameter than that of the granular carbide and activating the resultant mixture or compounding the granular active carbon in the spherical shape with the active carbon having the smaller grain diameter than that of the granular active carbon. The carbide in the spherical shape may be prepared by using a spherical resin as a raw material or may be obtained by polishing the pulverized carbide and having  $\leq 5$  aspect ratio. The pore volume can be regulated to  $\leq 0.55$  cc/g when the electrode is prepared from the active carbon according to the method for production. Thereby, the electric double layer capacitor of a high capacity is obtained.

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**CLAIMS**

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[Claim(s)]

[Claim 1] Activated carbon which blended at least two or more sorts of aspect ratios 1-5, activated carbon (A) with a mean particle diameter of 4-40 micrometers, and activated carbon (B) with a mean particle diameter of 4 micrometers or less.

[Claim 2] Activated carbon 10-60 mass % Containing activated carbon with a mean particle diameter [ of B ] of 4 micrometers or less according to claim 1.

[Claim 3] Activated carbon according to claim 1 or 2 whose tapping bulk density is 0.5-1.2g/cc.

[Claim 4] Activated carbon according to claim 3 whose pore volume is 0.5 - 1.5 cc/g.

[Claim 5] this -- claims 1-4 whose activated carbon particles of A are globular forms-like -- either -- the activated carbon of a publication.

[Claim 6] The manufacture approach of the activated carbon characterized by blending and carrying out activation of globular form granular carbide and the carbide with a particle size smaller than it.

[Claim 7] The manufacture approach of the activated carbon characterized by blending another grinding activated carbon with this for globular form granular carbide after activation.

[Claim 8] The manufacture approach of the activated carbon according to claim 6 or 7 obtained when this globular form granular carbide carries out carbonization baking of the globular form granular resin.

[Claim 9] The manufacture approach of the activated carbon according to claim 8 obtained when this globular form granular resin grinds and corns resin.

[Claim 10] The manufacture approach of the activated carbon characterized by blending the granular active carbon on a globular form, and activated carbon with a particle size smaller than it.

[Claim 11] The manufacture approach of the activated carbon according to claim 10 obtained when this globular form granular active carbon grinds and grinds activated carbon.

[Claim 12] a claim -- the electric double layer capacitor electrode which used the activated carbon of a publication as the main raw material one to 5 either.

[Claim 13] The electric double layer capacitor electrode according to claim 12 whose pore volume of the electrode when making it an electrode itself is 0.55 or less cc/g.

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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention offers the good activated carbon particle group of pack density, and relates to the electrode which made it the packed bed and used the object for porous electrodes, and its activated carbon with clearance of a contaminant, and shaping with the object for catalyst support, or binder resin.

[0002]

[Description of the Prior Art] Development of an environmental problem to an electric vehicle (EV) is prosperous these days. However, the sign to which EV of the actuation method of only a rechargeable battery still gets into stride from infrastructures, such as a charging equipment, not being fixed is not seen. HEV of the hybrid system which combined a conventional gasoline engine and a conventional mass capacitor more realistic than the bottom of such a situation is beginning to be commercialized prior to EV. This mass capacitor for HEV is called an electric double layer capacitor or a capacitor, and the miniaturization of a configuration and the increment in discharge capacity are demanded especially.

[0003] If an electric double layer capacitor is explained, it will become a basic configuration like drawing 1 . In drawing 1 , 1 is a polarizable electrode containing the electrolytic solution, and inter-electrode [ of a couple ] is divided with the separator 2 which consists of electric insulation material of ionic permeability. And the circumference of a polarizable electrode 1 is sealed with a sealing agent 3, and through the impermeable and conductive collecting electrode plate 4, the laminating of the a large number cel is carried out, and an activity is presented with this single cel.

[0004] Although activated carbon is generally used as a polarizable electrode, it is required for discharge capacity to be large as a property of the electrode. As a property that the electrode for raising discharge capacity should be held, about 10-20A micropore exists mostly in activated carbon, and it is so good that specific surface area is large. For this reason, carrying out processing which performs activation processing and increases pore is usually taken as the manufacture approach of activated carbon after calcinating carbon material.

[0005] That it is common using what a pitch and coconuts carbonize by baking, such as organic resin, as a raw material of activated carbon, it carries out elevated-temperature baking, grinding, and activation processing for these, and makes it activated carbon powder (grain) is . [0006] which is the process of activated carbon. This activated carbon adds PTFE for the carbon black for holding conductivity, and caking (polytetrafluoroethylene) etc., pokes it, is hardened, and becomes a tabular electrode plate. In order to raise the capacity of this electrode plate, it is required to raise, the surface area, i.e., the pore volume, of the whole electrode, but in order to raise pore volume, if it thinks with an isomorphism-like electrode plate, it is required to raise the specific surface area (surface area per weight) of activated carbon itself, or to raise the consistency (weight per volume) of an activated carbon electrode.

[0007] Usually activation is performed by the activated carbon production process as a means to raise specific surface area. About an activation art, the approach of the gas activation which lets the inside of a steam or carbon dioxide gas pass at an elevated temperature, and two kinds of approaches of the chemical activation which lets the inside of chemicals, such as lye, pass are common. In the semantics which raises specific surface area for high-capacity-izing, the chemical activation is more effective than gas activation.

[0008] It is raising the electrode consistency of an activated carbon electrode as another means of

high-capacity-izing. That is, about the ground activated carbon, although it was raising the bulk density at the time of restoration of the activated carbon in the case of making it an electrode (henceforth pack density), if a limitation was in pack density and specific surface area was raised in a certain particle size distribution, since pore would increase inside, when seeing by this volume as an electrode, there was dilemma that an electrode consistency fell.

[0009] Although it experimented in order to carry out various \*\*\*\* combination of the particle size distribution of the activated carbon ground so that this invention persons might make high density fill up with activated carbon and to raise pack density, the improvement in a consistency was not obtained.

[0010]

[Problem(s) to be Solved by the Invention] This invention offers the manufacture approach of the activated carbon which the bulk density when making it an electrode that it is easy to be filled up as activated carbon material, especially activated carbon material for electric double layer capacitor electrodes goes up and activated carbon, and the electrode whose capacity improved by using it.

[0011]

[Means for Solving the Problem] this invention persons were able to come to solve by the activated carbon particle group which controlled the process, configuration, and particle diameter which are described below, as a result of repeating research wholeheartedly about how the activated carbon which raised restoration nature has come out of the above-mentioned technical problem from the gestalt of the activated carbon. Namely, the manufacture approach of the activated carbon which mixes 1 globular form granular carbide and carbide with a particle size smaller than it, and is characterized by carrying out activation.

2) The manufacture approach of the activated carbon characterized by blending what ground the activated carbon which carried out activation of the globular form granular carbide independently after activation, and made particle size small.

3) The manufacture approach of the activated carbon characterized by blending globular form granular active carbon and activated carbon with a particle size smaller than it. It comes out. that an aspect ratio also uses five or less massive activated carbon cuts instead of aspect ratios being five or less massive carbide and globular form granular active carbon instead of globular form granular carbide. Although what kind of thing may be used when using resin as a raw material of the globular form granular carbide used for this process, it is using what was manufactured and sold as globular form granular resin, and globular form granular carbide as it is is obtained according to carbonization baking conditions. Moreover, even if globular form granular resin was not beforehand manufactured in the shape of a globular form, it may carry out carbonization baking of what the lump of resin was ground and corned and was made into the shape of a globular form. In any case, it can manufacture easily and cheaply by using phenol resin, guanamine resin, etc. Even if it does not use globular form granular carbide as a raw material, what made activated carbon the shape of a globular form by polish and grinding is sufficient as globular form activated carbon. Moreover, as for the approach of the activation in this manufacture, it is desirable to carry out by chemical activation by any activation approaches, in order to raise specific surface area, although it is possible.

[0012] Activated carbon which blended 4 aspect ratios 1-5, globular form activated carbon with a mean particle diameter of 4-40 micrometers, and activated carbon with a mean particle diameter of 4 micrometers or less as activated carbon in such a process.

- 5) Activated carbon given [ 10-60 mass % containing activated carbon with a mean particle diameter of 4 micrometers or less ] in four.
- 6) Activated carbon 4 whose tapping bulk density is 0.5-1.2g/cc, or given in five.
- 7) Activated carbon given in six given pore volume is 0.5 - 1.5 cc/g. \*\*\*\*\*. 8 these 4-7 [ moreover, ] -- the electric double layer capacitor electrode which used one of activated carbon. It was alike and the property of high capacity was acquired. Moreover, pore volume of the electrode at this time itself was able to be made into 0.55 or less cc/g.

[0013]

[Embodiment of the Invention] The gestalt of the activated carbon by this invention blends activated carbon with a mean particle diameter of 4 micrometers or less with aspect ratios 1-5 and activated carbon with a mean particle diameter of 4-40 micrometers. Moreover, the granular object or carbide of resin of an activated carbon raw material is blended beforehand, and that from which the presentation after activation becomes this combination after an appropriate time is also included. Generally it is expressed with the ratio of (the length of major axis)/(the length of a minor axis) an aspect ratio, and the value is calculated from a microphotography. [ of a particle ] Mean particle diameter is a path (D50 [ so-called ]) calculated from 50% of value of a cumulative-distribution curve.

[0014] In order to raise the pack density as an electrode, the configuration into which combination with the smaller powder bed with the magnitude which can trespass upon the opening of the powder bed which consists of large particle diameter further of particle diameter, and a particle tend to advance serves as an important element. Mixing a large particle group and a small particle group by the activated carbon of this invention and the configuration of a large particle are the points.

[0015] a large particle group -- aspect ratios 1-5 -- the shape of a globular form as used in the field of [ although as perfect at 1-2 a desirable ball (aspect ratio 1) as possible is desirable ] this invention -- the shape of a perfect circle-like ball, the shape of an ellipse, and a cocoon, and mass -- any are sufficient although the angle holds the globular form round further. Although especially the particle shape of a particle group with a mean particle diameter of 4 micrometers or less may not be specified and what kind of configuration is sufficient as it, its desirable spherical thing is good. The particle diameter of a big particle group has 4-40-micrometer preferably good 10-30 micrometers at mean particle diameter. Although it is not specified, when the opening for restoration of a small particle group is taken into consideration, the larger one of especially the minimum particle diameter in a big particle group than the mean particle diameter of a desirable small particle group is desirable. The particle diameter of a particle group small on the other hand is 4 micrometers or less in mean particle diameter, and is 1/5 or less [ of the path of a desirable big particle group ]. Although especially the minimum particle diameter is not specified, since trouble will be caused to an electrostatic-capacity manifestation in the electrode for electric double layer capacitors if particle diameter is too small, 0.5 micrometers or more are preferably good. Considering the restoration to the opening of a large particle group, below the mean particle diameter of a large particle group of the small diameter of grain of maximum size of a particle group is desirable.

[0016] although the loadings of the small particle group to a big particle group change according to mean particle diameter and particle size distribution -- ten to 60 mass % -- by making it desirable 20-40 mass % combination, slipping nature is improved, the opening of activated carbon is filled up with small activated carbon, and it was able to come to offer the activated

carbon particle group with a high tapping consistency. The opening which consists of big particle diameter cannot finish being filled up with 10% or less of combination, and the opening which consists of small particle diameter increases at 60% or more.

[0017] In addition, with tapping bulk density, weighing capacity of the 5.0g of the carbon powder is carried out, and it is put into the cell for measurement of 15mmphi, and sets in tapping equipment. The volume is measured, after it carries out drop height of 30mm, and a tapping rate in 0.2 seconds/time and they carry out free fall 10000 times. Let what calculated the consistency from the weight and the volume at that time be tapping bulk density. Moreover, the pore volume of activated carbon assumed that pore was filled up with Yuasa Ionics (AUTSORB-1MP mold) with liquid nitrogen, and was calculated from the capacity to which relative pressure stuck in the one neighborhood.

[0018] The point about the process of the invention in this application manufactures activated carbon by blending and carrying out activation of other carbide with a particle size smaller than it which was made by carrying out carbonization baking by others beforehand with globular form granular carbide. Or the activated carbon which carried out activation of the globular form granular carbide independently after activation is ground, and it manufactures by blending what made particle size small more. Globular form granular resin is used for globular form granular carbide as a start raw material, and it carries out carbonization baking of this, or may carry out carbonization baking of what used a grain and massive resin as globular form granular resin by grinding and the granulation.

[0019] Although chlorine-based resin, such as heat-curing resin, such as phenol resin, guanamine resin, xylene resin, furan resin, and melamine resin, and vinyl chloride, can be used for resin, globular form resin deposits a monomer in the state of emulsification or suspension by advancing a self-condensation reaction for a monomer in the state of emulsification or suspension, after advancing a self-condensation reaction by the homogeneous system. After an appropriate time, separation and the globular form granular resin after washing desiccation can be obtained for a solid from a solvent. Moreover, it is also possible to use the usual above-mentioned resin as globular form granular resin by grinding and the granulation. Grinding is good with the usual grinding equipments, such as a ball mill, a vibration mill, and jet mill grinding. Moreover, as for a granulation, stirring centrifugal granulation or spray dry is used.

[0020] It is more desirable to place the grain size of the above-mentioned globular form granular resin in accordance with the grain size of the activated carbon to be used. That is, it is important to hold the original globular form-like side for the grain of the resin of a basis baking and by carrying out activation to activated carbon in the form near a configuration as it is.

[0021] Although other carbide with this small particle size is good in addition if it is globular forms-like, it is not necessary to be a globular form-like, and is easy to be grinding and the thing which carried out activation after grinding or carbonization about usual resin after carbonization and activation. However, about the particle size, it is required to be a particle size smaller than globular form carbide. Extent of the magnitude of the particle size is desirable in order for about 20% or less of the particle size of globular form-like carbide to enter a clearance.

[0022] Since carbonization temperature has influence on a subsequent property, it is necessary to select the temperature optimal as activated carbon for capacitors, and generally 500 degrees C - about 1000 degrees C are usually used. When carbonization temperature is not much low, carbonizing becomes inadequate and subsequent activation serves as instability. If carbonization temperature is high, a surface functional group will decrease and it will activation-progress-come

to be hard. As carbonization temperature, 600-800 degrees C is desirable.

[0023] Although what kind of activation approach is sufficient as the activation after carbonization, in order to raise specific surface area, its chemical activation by caustic potash (KOH), caustic alkali of sodium (NaOH), the zinc chloride ( $\text{ZnCl}_2$ ), a phosphoric acid, etc. is desirable. Temperature at the time of activation is performed at 500-1000 degrees C. If activation temperature is low, micropore will increase, if activation temperature is high, meso holes will increase in number, and the activation temperature suitable for an application is selected.

[0024] Furthermore, it turned out that there is the same effectiveness as the above also by the activated carbon obtained by globular form granular active carbon's grinding the point of the process of the invention in this application, and corning. That is, even if it manufactured usual resin for carbonization, the activated carbon which carried out activation or coconut husk activated carbon, corks system activated carbon, etc., and it manufactured globular form activated carbon for usual activated carbon in grinding, granulation or polish, grinding, etc. and having been blended with other activated carbon with this small particle size, it turned out that the activated carbon particle group whose pack density improved like the above has come out. Usually, a ball mill, a vibration mill, a jet mill, etc. grind activated carbon to 2-8 micrometers preferably the mean particle diameter of 0.5-10 micrometers, and it is preferably corned to 15-30 micrometers the mean particle diameter of 4-40 micrometers by the stirring centrifugal granulating machine, spray dry, etc. The need is accepted, and it grinds and grinds with a wet media mill etc.

[0025] The activated carbon particle group whose pack density improved can be offered by blending particle size with small large globular form particle size and this particle size, as stated so far. As stated previously, it is not necessary to be globular form activated carbon the ball of a perfect perfect circle, and there should just be about [ 1-5 ] by the aspect ratio. It is 1-2 preferably. Moreover, as an electrode for cells, the mean particle diameter of 4-40 micrometers is desirable massive, and globular form activated carbon has good 15-30 micrometers. As for the activated carbon of a particle size smaller than it which is blended together, 1/5 or less [ of the large mean particle diameter of activated carbon desirable the mean particle diameter of 4 micrometers or less ] is desirable although between globular form particles is filled. In addition, although it does not need to be a globular form-like, if this small activated carbon is a globular form-like, it is good for restoration. Activated carbon with a small particle size [ this ] of 4 micrometers or less is desirable in order that ten to 60 whole mass % and carrying out 20-40 mass % extent combination preferably may raise the consistency at the time of restoration. According to the manufacturing method of this invention, the activated carbon which blended aspect ratios 1-5, with a mean particle diameter [ 4-40micro ] globular form-like activated carbon, and activated carbon with a mean particle diameter of 4 micrometers or less was obtained, and while the tapping bulk density was as high as 0.5 - 1.2 cc/g, the activated carbon with which pore volume has pore in 0.5 - 1.5 cc/g was obtained.

[0026] Using this activated carbon, Teflon was used as the binder, and it fabricated in the shape of a sheet with rolling, and considered as the electrode sheet stuck to the stainless steel network by pressure, and when constant current charge and discharge were performed with the configuration of the electric double layer capacitor described previously and the capacity at the time of discharge was measured, engine performance sufficient as an electric double layer was obtained. Moreover, when the pore volume of the electrode at this time itself was measured in the mercury porosimeter (Yuasa Ionics make: pore master 33), it was the opening of 0.55 or less

cc/g.

[0027]

[Example] Hereafter, explanation about the electrode which used the activated carbon manufacture approach of this invention and its activated carbon, and it according to the example is performed.

(Adjustment of the globular form-like resin by the synthesis method) In order to obtain a globular form phenol resin particle, phenol 200g, 37% formalin 200g, and 64.2g of 28% aqueous ammonia were thrown in, and it considered as the uniform solution. Made it react, and filtration-rinsed, this solution was made to have added and to dry 5.8g of potassium fluorides, 8.4g of calcium chlorides, and dispersion liquid of 40g of water under stirring, and the globular form-like phenol resin particle with a mean particle diameter of 30 micrometers was obtained. (Globular form-like resin of Type A)

(Globular form-like carbide from synthesis method globular form-like resin) The globular form-like resin of the above-mentioned type A was carbonized at 600 degrees C among nitrogen-gas-atmosphere mind for 2 hours. The obtained carbon powder was 25 micrometers in mean particle diameter, and the aspect ratio was 1. (Globular form-like carbide of Type a)

(Globular form-like carbide by the processing method) Commercial phenol resin (Gunei Chemical Industry PL-2211) was stiffened at 180 degrees C, and it carbonized at 600 degrees C. After grinding to 2 micrometers by the vibration ball mill, it corned with the granulating machine of trade name:FUKAE3 L type, the wet-grinding machine (trade name: DYNO-MILL LDLA mold) adjusted if needed, and globular form carbide was obtained. This was carbonized at 600 degrees C among nitrogen-gas-atmosphere mind for 2 hours. The mean particle diameter of 23 micrometers and an aspect ratio were 2. (Globular form-like carbide of Type b)

(Carbide of a small particle size) Commercial phenol resin (Gunei Chemical Industry PL-2211) was stiffened at 180 degrees C, and it carbonized at 600 degrees C. Then, it ground by the vibration ball mill and adjusted to the mean particle diameter of 4 micrometers.

[0028] [Example 1] Caustic potash (KOH) was fed into the globular form-like carbide of Type a 2.5 times, and reaction activation was carried out at 700 degrees C among nitrogen-gas-atmosphere mind. It dried, after rinsing well and removing K. The activated carbon which the mean particle diameter after activation does not change to 25 micrometers and an aspect ratio 1 was obtained. Activation of the carbide of said small particle size was carried out on these conditions, and activated carbon with a mean particle diameter of 3.8 micrometers was obtained. This globular form activated carbon and the activated carbon of a small particle size were blended with the weight ratio 65:35, and were tapped. The tapping bulk density at this time and the pore volume of activated carbon were measured. This result is shown in a table 1.

[0029] Next, 9 % of the weight of furnace black was added for Teflon powder to this activated carbon 10% of the weight, and it mixed with the agate mortar, fabricated with the rolling roller in the shape of a sheet, and was stuck to stainless steel net producing by pressure, and the electrode sheet was created. This sheet was cut to 10x10mm, and it considered as the positive electrode and the electrode for negative electrodes. An electrode is set to aluminum cel through the separator made from 1mm glass filter in between, and they are 4 (C<sub>2</sub>H<sub>5</sub>) and NBF<sub>4</sub> to propylene carbonate 1L. It considered as the electrolytic solution, and was immersed and the liquid which dissolved 1 mol/L was used as the electric double layer capacitor. Constant current charge and discharge were performed to this capacitor in current density 1.0 mA/cm<sup>2</sup> and the electrical-potential-difference range 0-2.3V, and capacity was measured at the time of discharge. The bulk



density (electrode consistency) of the activated carbon as this electrode, and the pore volume and discharge capacity of an electrode are shown in a table 1.

[0030] Chemical activation was performed for [example 2] type b, globular form carbide on the same conditions as an example 1, and an aspect ratio 2 and globular form-like activated carbon with a mean particle diameter of 23 micrometers were obtained. This and the carbide of said small particle size were blended with what carried out activation according to these conditions 60:40, and were tapped. The tapping bulk density at this time and pore volume were measured. This result is shown in a table 1. By the same technique as an example 1, the electrode was created using this activated carbon and constant current charge and discharge were performed as an electric double layer capacitor. The result of having measured an electrode consistency and discharge capacity similarly is shown in a table 1.

[0031] [Example 3] marketing phenol resin (Gunei Chemical Industry PL-2211) was stiffened at 180 degrees C, and it carbonized at 600 degrees C. Caustic potash (KOH) was thrown into this 2.5 times, and reaction activation was carried out at 700 degrees C among nitrogen-gas-atmosphere mind. It dried, after rinsing well and removing K. next, this -- trade name: -- FUKAE It corned with the granulating machine of 3 L types, the wet-grinding machine (trade name: DYNO-MILL LDLA mold) adjusted if needed, and the mean particle diameter of 30 micrometers and the globular form activated carbon of an aspect ratio 3 were obtained. Activated carbon with a mean particle diameter of 3.8 micrometers which could carry out activation of this and the carbide of said small particle size on these conditions was blended and tapped to 65:35. The tapping bulk density at this time and pore volume were measured. This result is shown in a table 1. Below, like the example 1, the electrode was created, the electric double layer capacitor was constructed, and same measurement was performed. This result is shown in a table 1.

[0032] [Example 4] type a globular form-like carbide and the carbide of said small particle size were blended with 60:40, the amount charge of the caustic potash (KOH) was carried out 2.5 times, and activation was carried out at 700 degrees C in nitrogen. Then, it rinsed, and it dried, after removing K. Hereafter, like the example 1, the electrode was created, the electric double layer capacitor was constructed, and same measurement was performed. This result is shown in a table 1.

[0033] [Example 1 of comparison] marketing phenol resin (Gunei Chemical Industry P1-2211) was stiffened at 180 degrees C, it carbonized at 600 degrees C, KOH of an amount was supplied 2.5 times, and activation was performed at 700 degrees C among nitrogen-gas-atmosphere mind. Then, it ground in mean particle diameter of 20 micrometers by the vibration ball mill. The aspect ratio was set to 6 and square flat activated carbon was obtained. The activated carbon of a small particle size used for this and an example 1 was blended with 65:35, and the electrode was created on the same conditions as an example 1, the electric double layer capacitor was constructed, and each measurement was performed. This result is shown in a table 1.

[0034] [Example 2 of comparison] marketing phenol resin (Gunei Chemical Industry P1-2211) was stiffened at 180 degrees C, it carbonized at 600 degrees C, KOH of an amount was supplied 2.5 times, and activation was performed at 700 degrees C among nitrogen-gas-atmosphere mind. Then, it ground in mean particle diameter of 30 micrometers by the vibration ball mill. The aspect ratio was set to 5 and square flat activated carbon was obtained. The activated carbon of a small particle size used for this and an example 1 was blended with 60:40, and the electrode was created on the same conditions as an example 1, the electric double layer capacitor was constructed, and each measurement was performed. This result is shown in a table 1.

[0035]

[Effect of the Invention] By the approach of this invention, pore volume can be increased by raising the pack density of activated carbon, the electrode which used the activated carbon manufactured by this process has a high electrode consistency, and the discharge capacity of high performance is obtained.

[A table 1]

[0036]

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## DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is the basic block diagram of an electric double layer capacitor.

[Description of Notations]

1 Activated Carbon Electrode

2 Porous Glass Separator

3 Packing Sheet

4 Collecting Electrode Plate

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(54) 【発明の名称】 活性炭及びその製法

(57) 【要約】

【課題】活性炭素材として充填性のよい、電極にした時の高密度が上がる活性炭の製造方法及び活性炭、それを使用した容量の向上した電気二重層コンデンサ用電極を提供する。

【解決手段】球形状の粒状炭化物和それより粒径の小さな炭化物を混合し賦活する、あるいは球形状の粒状活性炭とそれより粒径の小さな活性炭を配合する。球形状の炭化物は、球形状の樹脂を原料としてもよいし、粉碎した炭化物を研磨したものでアスペクト比5以下のものでも良い。この製法の活性炭にて電極を作成したとき、細孔容積を0.55cc/g以下にすることが出来、高容量の電気二重層コンデンサが得られた。

## 【特許請求の範囲】

【請求項1】アスペクト比1～5、平均粒径4～40  $\mu\text{m}$ の活性炭(A)と平均粒径4  $\mu\text{m}$ 以下の活性炭(B)の少なくとも2種以上を配合した活性炭。

【請求項2】Bの平均粒径4  $\mu\text{m}$ 以下の活性炭を10～60質量%含む請求項1記載の活性炭。

【請求項3】タッピング嵩密度が0.5～1.2 g/ccである請求項1又は2記載の活性炭。

【請求項4】細孔容積が0.5～1.5 cc/gである請求項3記載の活性炭。

【請求項5】該Aの活性炭粒子が球形状である請求項1～4いずれか記載の活性炭。

【請求項6】球形状の粒状炭化物とそれより粒径の小さな炭化物を配合し、賦活することを特徴とする活性炭の製造方法。

【請求項7】球形状の粒状炭化物を賦活後、これに別の粉砕活性炭を配合することを特徴とする活性炭の製造方法。

【請求項8】該球形状の粒状炭化物が球形状の粒状樹脂を炭化焼成することにより得られたものである請求項6又は7記載の活性炭の製造方法。

【請求項9】該球形状の粒状樹脂が樹脂を粉砕、造粒することにより得られたものである請求項8記載の活性炭の製造方法。

【請求項10】球形上の粒状活性炭とそれより粒径の小さな活性炭を配合することを特徴とする活性炭の製造方法。

【請求項11】該球形状の粒状活性炭が活性炭を研磨、摩砕することにより得られたものである請求項10記載の活性炭の製造方法。

【請求項12】請求項1～5いずれか記載の活性炭を主原料とした電気二重層コンデンサ電極。

【請求項13】電極にした時の電極自体の細孔容積が0.55 cc/g以下である請求項12記載の電気二重層コンデンサ電極。

## 【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、充填密度の良好な活性炭粒子群を提供するもので、充填層にして汚染物質の除去や、触媒担持用あるいはバインダー樹脂との成形で多孔質電極用また、その活性炭を使用した電極に関するものである。

【0002】

【従来の技術】昨今環境問題から電気自動車(EV)の開発が盛んである。しかし、二次電池のみの駆動方式のEVは充電設備等のインフラが整備されていないことから未だ本格化する兆しが見られていない。このような状況下より現実的な従来のガソリンエンジンと大容量コンデンサを組み合わせたハイブリッド方式のHEVがEVに先駆けて商品化され始めている。このHEV用大容量

コンデンサは電気二重層コンデンサあるいはキャパシタと呼ばれるものであり、特に形状の小型化、放電容量の増加が要求されている。

【0003】電気二重層コンデンサについて説明すると、図1のような基本構成となる。図1において1は電解液を含む分極性電極であり、一對の電極間はイオン透過性の電気絶縁材からなるセパレーター2で仕切られている。そして分極性電極1の周辺は封止材3で密閉され、またこの単セルは不浸透性かつ導電性の集電板4を介して多数セルが積層されて使用に供される。

【0004】分極性電極として一般には活性炭が使用されるが、その電極の特性として放電容量が大きいために必要である。放電容量を上げるための電極の保持すべき特性としては、活性炭素内に10～20 A程度の微細孔が多く存在し比表面積が大きいほど良好である。このために炭素材を焼成後、賦活処理を行い細孔を増やす処理をすることが活性炭の製造方法として通常とられている。

【0005】活性炭の原料としては、ピッチ、椰子から、有機樹脂等、焼成により炭化するものを使用するのが一般的であり、これらを高温焼成、粉砕、賦活処理をし、活性炭粉(粒)にするのが活性炭の製法である。

【0006】この活性炭は、導電性を保持するためのカーボンブラック、粘結のためのPTFE(ポリテトラフルオロエチレン)等を添加し、突き固められ板状の電極板になる。この電極板の容量を上げるため、電極全体の表面積すなわち細孔容積を上げることが必要であるが、細孔容積を上げるためには、同形状の電極板で考えれば活性炭自体の比表面積(重量あたりの表面積)を上げるか、活性炭電極の密度(体積あたりの重量)をあげる必要がある。

【0007】比表面積をあげる手段として活性炭製造工程で賦活を行うことが普通である。賦活処理方法については、高温で水蒸気中、あるいは炭酸ガス中を通すガス賦活の方法と、アルカリ液等の薬品中を通す薬品賦活の2通りの方法が一般的である。高容量化のため比表面積を上げる意味では、薬品賦活のほうがガス賦活より効果的である。

【0008】高容量化のもう一つの手段としては活性炭電極の電極密度を上げることである。すなわち電極にする場合の活性炭の充填時の嵩密度(以下充填密度という。)を上げることであるが、粉砕された活性炭については、ある粒径分布では充填密度に限界があり、また比表面積を上げていくと、細孔が内部に増えるため、電極として同体積で見れば電極密度が下がるというジレンマがあった。

【0009】本発明者らは、活性炭を少しでも高密度に充填させるべく、粉砕された活性炭の粒度分布を各種変え配合し充填密度を上げるべく実験を行ったが密度の向上は得られなかった。

【0010】

【発明が解決しようとする課題】本発明は、活性炭素材特に電気二重層コンデンサ電極用の活性炭素材として充填しやすく電極にしたときの高密度が上がる活性炭の製造方法及び活性炭、それを使用することにより容量の向上した電極を提供するものである。

【0011】

【課題を解決するための手段】本発明者らは、上記課題をその活性炭の形態から如何に充填性を高めた活性炭が出来るかについて鋭意研究を重ねた結果、以下に述べる製法及び形状及び粒子径を制御した活性炭粒子群で解決できるに至った。すなわち

1) 球形状の粒状炭化物和それより粒径の小さな炭化物を混合し、賦活することを特徴とする活性炭の製造方法。

2) 球形状の粒状炭化物を賦活後、別に賦活した活性炭を粉砕し粒径を小さくしたものを配合することを特徴とする活性炭の製造方法。

3) 球形状の粒状活性炭とそれより粒径の小さな活性炭を配合することを特徴とする活性炭の製造方法。である。球形状の粒状炭化物のかわりにアスペクト比が5以下の塊状の炭化物、球形状の粒状活性炭のかわりにアスペクト比が5以下の塊状の活性炭も使用することがきる。この製法に使用する球形状の粒状炭化物の原料として樹脂を使用する場合は、いかなるものでも良いが、球形状の粒状樹脂として製造、販売されたものを使用することで、炭化焼成条件によりそのままの球形状の粒状炭化物が得られる。また、球形状の粒状樹脂は、あらかじめ球形状に製造されたものでなくても、樹脂の塊を粉砕、造粒して球形状にしたものを炭化焼成してもよい。いずれの場合でも、フェノール樹脂、グアニミン樹脂等を使用することにより、容易にかつ安価に製造できる。球形状の活性炭は、球形状の粒状炭化物を原料としなくとも活性炭を研磨、摩砕により球形状にしたものでもかまわない。またこの製造での賦活の方法は、いかなる賦活方法でも可能であるが、比表面積を上げるためには、薬品賦活で行うことが好ましい。

【0012】このような製法にて活性炭として、

4) アスペクト比1~5、平均粒径4~40 $\mu$ mの球形状の活性炭と平均粒径4 $\mu$ m以下の活性炭を配合した活性炭。

5) 平均粒径4 $\mu$ m以下の活性炭を10~80質量%含む4)記載の活性炭。

6) タッピング嵩密度が0.5~1.2g/ccである4)又は5)記載の活性炭。

7) 細孔容積が0.5~1.5cc/gである8)記載の活性炭。が得られた。また、

8) これら4)~7)いずれかの活性炭を使用した電気二重層コンデンサ電極。にて高容量の特性が得られた。またこの時の電極自体の細孔容積は、0.55cc/g

以下にすることができた。

【0013】

【発明の実施の形態】本発明による活性炭の形態は、アスペクト比1~5、平均粒径4~40 $\mu$ mの活性炭に平均粒径4 $\mu$ m以下の活性炭を配合したものである。また、活性炭原料の樹脂の粒状物又は炭化物があらかじめ配合され、しかる後に賦活後の組成がこの配合になるものも含む。アスペクト比とは一般に粒子の(長軸の長さ)/(短軸の長さ)の比で表され、その値は顕微鏡写真から求められる。平均粒子径とは、累積分布曲線の50%の値から求められた径(いわゆるD50)である。

【0014】電極としての充填密度をあげるためには、大きい粒子径で構成される粉体層の空隙に更に侵入できる大きさを持った、より小さな粒子径の粉体層との組合せ、粒子の進入し易い形状が重要な要素となる。本発明の活性炭では、大きい粒子群と小さい粒子群を混合することと、大きい粒子の形状がポイントである。

【0015】大きい粒子群は、アスペクト比1~5好ましくは1~2でなるべく完全な球(アスペクト比1)が好ましいが、本発明でいう球形状とは真円状の球、楕円状あるいは蘭状、塊状、更には角が丸く球形を保持しているもののいずれでもよい。平均粒径4 $\mu$ m以下の粒子群の粒子形状は特に規定されるものではなく、いかなる形状でもよいが好ましくは球状のものがよい。大きな粒子群の粒子径は平均粒径で4~40 $\mu$ m好ましくは10~30 $\mu$ mがよい。大きな粒子群中の最小粒子径は、特に規定されないが、小さな粒子群の充填のための空隙を考慮すると好ましくは小さな粒子群の平均粒径より大きいほうが好ましい。一方小さな粒子群の粒子径は、平均粒径4 $\mu$ m以下であり、好ましくは大きな粒子群の径の1/5以下である。最小粒子径は特に規定されないが、電気二重層コンデンサ用の電極では、粒子径が小さすぎると静電容量発現に支障をきたすので、好ましくは0.5 $\mu$ m以上がよい。小さな粒子群の最大粒子径は、大きい粒子群の空隙への充填を考えると、大きい粒子群の平均粒径以下が好ましい。

【0016】大きな粒子群への小さな粒子群の配合量は、平均粒子径、粒度分布により変わるが、10~80質量%好ましくは20~40質量%配合することにより、滑り性が改善され活性炭同士の間隙に小さな活性炭が充填され、タッピング密度の高い活性炭粒子群を提供できるに至った。10%以下の配合では、大きな粒子径で構成される空隙を埋めきれず、60%以上では、小さい粒子径で構成される空隙が多くなる。

【0017】なお、タッピング嵩密度とは、炭素粉末5.0gを秤量し、15mm $\phi$ の測定用セルに入れ、タッピング装置にセットする。落下高さ30mm、タッピング速度を0.2秒/回とし10000回自由落下させた後、その体積を測定する。その時の重量と体積から密度を計算したものをタッピング嵩密度とする。また、活

性炭の細孔容積は、ユアサイオニクス(AUTSORB-1MP型)にて細孔が液体窒素により充填されていると仮定し、相対圧力が1付近で吸着したガス量から求めた。

【0018】本願発明の製法についてのポイントは、球形状の粒状炭化物とあらかじめ他で炭化焼成し作られたそれより粒径の小さな他の炭化物を配合し、賦活することにより活性炭を製造するものである。あるいは、球形状の粒状炭化物を賦活後、別に賦活した活性炭を粉砕し、より粒径を小さくしたものを配合することにより製造するものである。球形状の粒状炭化物は、出発原料として球形状の粒状樹脂を使用し、これを炭化焼成する、あるいは粒、塊状樹脂を粉砕、造粒により球形状の粒状樹脂にしたものを炭化焼成してもよい。

【0019】樹脂は、フェノール樹脂、グアニミン樹脂、キシレン樹脂、フラン樹脂、メラミン樹脂等の熱硬化樹脂、塩化ビニール等の塩素系樹脂が使用できるが、球形状の樹脂はモノマーを乳化あるいは懸濁状態で自己縮合反応を進めるか、あるいはモノマーを均一系で自己縮合反応を進めた後、乳化あるいは懸濁状態で析出させる。しかる後、溶媒から固形物を分離、洗浄乾燥後球形状の粒状樹脂を得ることができる。また、通常の上記樹脂を粉砕、造粒により球形状の粒状樹脂にすることも可能である。粉砕はボールミル、振動ミル、ジェットミル粉砕等通常の粉砕装置で良い。又、造粒は攪拌連心造粒あるいはスプレードライ等が使用される。

【0020】上記球形状の粒状樹脂の粒度は、使用する活性炭の粒度にあわせて置いたほうが好ましい。すなわち、もとの樹脂の粒をそのままの形状に近い形で活性炭に焼成、賦活することにより元の球形状面を保持することが大事である。

【0021】該粒径の小さな他の炭化物は球形状であればなお良いが、球形状である必要がなく、通常の樹脂を炭化、賦活後、粉砕あるいは炭化後に粉砕、賦活したもので良い。ただし、その粒径については球形状の炭化物よりも小さい粒径であることが必要である。その粒径の大きさの程度は、球形状炭化物の粒径の約20%以下が隙間に入り込むためには好ましい。

【0022】炭化温度は、その後の特性に影響があるためコンデンサ用活性炭として最適な温度を選定する必要があり、通常は500℃～1000℃程度が一般に用いられる。炭化温度があまり低い場合、炭化が不十分となり、その後の賦活が不安定となる。炭化温度が高いと表面官能基が少なくなり賦活進み難くなる。炭化温度としては、800～800℃が好ましい。

【0023】炭化後の賦活は、いかなる賦活方法でもよいが、比表面積をあげるためには苛性カリ(KOH)、苛性ソーダ(NaOH)、塩化亜鉛(ZnCl<sub>2</sub>)、リン酸等による薬品賦活が好ましい。賦活時の温度は、500～1000℃で行われる。賦活温度が低いとミクロ

孔が多くなり、賦活温度が高いとメソ孔が多くなり、用途に合った賦活温度が選定される。

【0024】更に本願発明の製法のポイントは、球形状の粒状活性炭が粉砕、造粒することにより得られた活性炭でも上記と同様の効果がある事がわかった。すなわち、通常の樹脂を炭化、賦活した活性炭あるいは椰子ガラ活性炭、コークス系活性炭等、通常の活性炭を粉砕、造粒あるいは研磨、摩砕等にて球形状の活性炭を製造し、該粒径の小さな他の活性炭と配合しても上記と同様に充填密度の向上した活性炭粒子群が出ることがわかった。通常活性炭を、ボールミル、振動ミル、ジェットミル等で平均粒径0.5～10μm、好ましくは2～8μmに粉砕し、攪拌連心造粒機、スプレードライ等で平均粒径4～40μm、好ましくは15～30μmに造粒する。必要に応じて湿式メディアミル等で研磨、摩砕する。

【0025】これまで述べてきたように球形状の大きい粒径と該粒径の小さい粒径を配合することにより充填密度の向上した活性炭粒子群を提供することができる。先に述べたように、球形状の活性炭は、完全な真円の球である必要はなく、アスペクト比で1～5程度であればよい。好ましくは1～2である。また電池用の電極としては、塊状又は球形状の活性炭は平均粒径4～40μmが、好ましくは15～30μmがよい。一緒に配合するそれより小さい粒径の活性炭は平均粒径4μm以下好ましくは大きい活性炭の平均粒径の1/5以下が、球形状の粒子間を埋めるのに好ましい。なお、この小さい活性炭は、球形状である必要はないが、球形状であればなお充填のためには良い。この4μm以下の小さな粒径の活性炭は、全体の10～60質量%、好ましくは20～40質量%程度配合することが充填時の密度をあげるために好ましい。本発明の製造法により、アスペクト比1～5、平均粒径4～40μmの球形状活性炭と平均粒径4μm以下の活性炭を配合した活性炭が得られ、そのタッピング密度は0.5～1.2cc/gと高いとともに、細孔容積は、0.5～1.5cc/gに細孔を持つ活性炭が得られた。

【0026】この活性炭を用いて、テフロンをバインダーとし、圧延によりシート状に成形し、ステンレス網に圧着した電極シートとし、先に述べた電気二重層コンデンサの構成にて定電流充放電を行い放電時の容量を測定したところ電気二重層として十分な性能が得られた。又、この時の電極自体の細孔容積は、水銀ポロシメーター(ユアサイオニクス製：ポアマスター33)にて測定したところ、0.55cc/g以下の空隙であった。

【0027】

【実施例】以下、実施例により本発明の活性炭製造方法、及びその活性炭、それを使用した電極についての説明をおこなう。

(合成法による球形状樹脂の調整) 球形状のフェノール

樹脂粒子を得るため、フェノール200g、37%ホルマリン200g、28%アンモニア水84.2gを投入し均一な溶液とした。この溶液に攪拌下で、弗化カリウム5.8g、塩化カルシウム8.4g、水40gの分散液を添加し、反応させ濾過水洗、乾燥させ平均粒径30 $\mu$ mの球形状フェノール樹脂粒子を得た。(タイプAの球形状樹脂)

(合成法球形状樹脂からの球形状炭化物) 上記タイプAの球形状樹脂を窒素雰囲気中600℃で2時間炭化した。得られた炭素粉は平均粒径25 $\mu$ m、アスペクト比は1であった。(タイプaの球形状炭化物)

(加工法による球形状炭化物) 市販フェノール樹脂(群栄化学PL-2211)を180℃で硬化させ、600℃で炭化した。振動ボールミルで2 $\mu$ mに粉碎した後、商品名:FUKAE 3L型の造粒機にて造粒し、必要に応じ湿式粉碎器(商品名:DYNO-MILL LDLA型)にて調整し、球形状の炭化物を得た。これを窒素雰囲気中600℃で2時間炭化した。平均粒径23 $\mu$ m、アスペクト比は2であった。(タイプbの球形状炭化物)

(小さい粒径の炭化物) 市販フェノール樹脂(群栄化学PL-2211)を180℃で硬化させ、600℃で炭化した。その後、振動ボールミルで粉碎し、平均粒径4 $\mu$ mまで調整した。

【0028】[実施例1] タイプaの球形状炭化物に苛性カリ(KOH)を2.5倍投入し、窒素雰囲気中700℃で反応賦活した。良く水洗し、Kを除去した後、乾燥した。賦活後の平均粒径は25 $\mu$ m、アスペクト比1と変わらない活性炭が得られた。前記小さい粒径の炭化物を同条件で賦活し、平均粒径3.8 $\mu$ mの活性炭を得た。この球形状の活性炭と小さい粒径の活性炭を重量比65:35に配合しタッピングした。この時のタッピング嵩密度、活性炭の細孔容積を測定した。この結果を表1に示す。

【0029】次にこの活性炭にテフロン粉末を10重量%、ファーンブラック9重量%を加えメノウ乳鉢で混合し、圧延ローラーにてシート状に成形し、ステンレス製網に圧着し電極シートを作成した。このシートを10 $\times$ 10mmに切り正極及び負極用電極とした。電極を、1mmガラスフィルター製セパレータを間に介してA1セルにセットしプロピレンカーボネート1Lに(C<sub>3</sub>H<sub>8</sub>)<sub>2</sub>NBF<sub>4</sub> 1mol/Lを溶解した液を電解液とし浸漬し、電気二重層コンデンサとした。このコンデンサに電流密度1.0mA/cm<sub>2</sub>、電圧範囲0~2.3Vで定電流充放電を行い放電時において容量を測定した。この電極としての活性炭の嵩密度(電極密度)及び電極の細孔容積と放電容量を表1に示す。

【0030】[実施例2] タイプbの球形状の炭化物を実施例1同様の条件で薬品賦活を行いアスペクト比2、平均粒径23 $\mu$ mの球形状活性炭を得た。これと、前記

小さい粒径の炭化物を同条件により賦活したものと60:40に配合しタッピングした。この時のタッピング嵩密度、細孔容積を測定した。この結果を表1に示す。実施例1と同様の手法により、この活性炭を用いて電極を作成し、電気二重層コンデンサとして定電流充放電を行った。同様に電極密度、放電容量を測定した結果を表1に示す。

【0031】[実施例3] 市販フェノール樹脂(群栄化学PL-2211)を180℃で硬化させ、600℃で炭化した。これに苛性カリ(KOH)を2.5倍投入し、窒素雰囲気中700℃で反応賦活した。良く水洗し、Kを除去した後、乾燥した。次にこれを商品名:FUKAE 3L型の造粒機にて造粒し、必要に応じ湿式粉碎器(商品名:DYNO-MILL LDLA型)にて調整し、平均粒径30 $\mu$ m、アスペクト比3の球形状の活性炭を得た。これと、前記小さい粒径の炭化物を同条件で賦活し得た平均粒径3.8 $\mu$ mの活性炭を65:35に配合し、タッピングした。この時のタッピング嵩密度、細孔容積を測定した。この結果を表1に示す。以下実施例1と同様に、電極を作成、電気二重層コンデンサを組んで同様の測定を行った。この結果を表1に示す。

【0032】[実施例4] タイプaの球形状炭化物と前記小さい粒径の炭化物を60:40に配合し、苛性カリ(KOH)を2.5倍量投入し窒素中で700℃にて賦活した。その後、水洗し、Kを除去した後乾燥した。以下、実施例1同様に、電極を作成、電気二重層コンデンサを組んで同様の測定を行った。この結果を表1に示す。

【0033】[比較例1] 市販フェノール樹脂(群栄化学P1-2211)を180℃で硬化させ、600℃で炭化し、2.5倍量のKOHを投入し、窒素雰囲気中700℃で賦活を行った。その後、振動ボールミルで平均粒径20 $\mu$ mに粉碎した。アスペクト比は6となり、角張った扁平な活性炭が得られた。これと実施例1に使用した小さな粒径の活性炭を65:35に配合し、また実施例1同様の条件にて電極を作成、電気二重層コンデンサを組み各測定を行った。この結果を表1に示す。

【0034】[比較例2] 市販フェノール樹脂(群栄化学P1-2211)を180℃で硬化させ、600℃で炭化し、2.5倍量のKOHを投入し、窒素雰囲気中700℃で賦活を行った。その後、振動ボールミルで平均粒径30 $\mu$ mに粉碎した。アスペクト比は5となり、角張った扁平な活性炭が得られた。これと実施例1に使用した小さな粒径の活性炭を60:40に配合し、また実施例1同様の条件にて電極を作成、電気二重層コンデンサを組み各測定を行った。この結果を表1に示す。

【0035】

【発明の効果】本発明の方法により、活性炭の充填密度を上げることにより、細孔容積を増すことができ、本製

法により製造した活性炭を使用した電極は電極密度が高 \*【表1】

く、高性能の放電容量が得られる。

\*

	細孔容積 (活性炭)	タッピング 濃密度	電極密度	細孔容積 (電極)	放電容量	備考:球形状の粒状活性炭		
	cc/g	g/cc	g/cc	cc/g	F/cc	アスペクト 比	平均粒径 μm	配合比 重量%
実施例1	1.0	0.70	0.89	0.43	31	1	25	65
実施例2	1.1	0.87	0.88	0.46	30	2	23	60
実施例3	1.2	0.85	0.84	0.50	29	3	30	65
実施例4	1.0	0.65	0.88	0.44	29	—	—	—
比較例1	1.1	0.40	0.55	0.63	23	—	—	—
比較例2	1.0	0.42	0.58	0.61	24	—	—	—

【0036】

10※ 1 活性炭電極

【図面の簡単な説明】

2 多孔質ガラスセパレーター

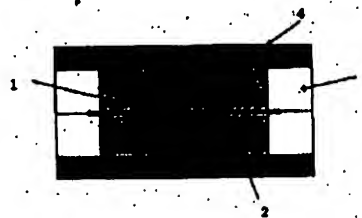
【図1】電気二重層コンデンサの基本構成図である。

3 パッキンシート

【符号の説明】

※ 4 集電板

【図1】



フロントページの続き

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